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(2 pages total)

METHOD FOR MANUFACTURING METALLIC TITANIUM BY ELECTRICAL DISCHARGE

Brief Description of the Drawings

The attached figure shows one example of the working of the method of the present invention, and is a longitudinal sectional view.

Detailed Description of the Invention

The present invention relates to a method for obtaining metallic titanium from titanium tetrachloride by an electrical discharge, which is characterized by the fact that titanium tetrachloride and hydrogen are caused to jet from one pole, and a discharge is caused to take place between this pole and the other pole, thus decomposing the titanium tetrachloride so that metallic titanium is produced. The object of the present invention is to provide a method for producing metallic titanium more economically and by means of an easier operation than in conventional methods, such as substitution methods using metallic magnesium.

The present invention is a method for obtaining metallic titanium by subjecting titanium tetrachloride to an electrical discharge; in this method, titanium tetrachloride and hydrogen are caused to jet from one pole, and a discharge is caused to take place between this pole and the other pole, thus decomposing the titanium tetrachloride so that metallic titanium is produced.

Conventionally, the manufacture of titanium tetrachloride by treating rutile, titanium iron ore or titanium dioxide, etc., with chlorine gas has been industrialized and widely used. Recently, furthermore, methods for producing metallic titanium from the titanium tetrachloride thus obtained have been particularly studied, and various methods have been proposed. A method that is currently being practiced industrially is a method in which titanium tetrachloride and metallic magnesium are reacted in a high-temperature inert gas, so that metallic titanium is produced by the following reaction:

$$TiCl_4 + 2Mg = Ti + 2MgCl_2$$

However, such a method requires that high-purity metallic magnesium be used at the rate of 1.1 to 1.5 times the amount of metallic titanium; furthermore, the apparatus is complicated, and an intermittent operation is ordinarily unavoidable. Various studies are being conducted in order to solve such problems; however, the method of the present invention eliminates the drawbacks of such conventional problems, and offers an extremely novel method for the manufacture of metallic titanium.

Specifically, noting that metallic titanium can be produced according to the following formula by reacting titanium chloride with hydrogen at a high temperature of 2000°C or higher, the present inventor conducted various studies, and perfected the present invention.

$TiCl_4 + 2H_2 = Ti + 4HCl$

However, it is extremely difficult to obtain metallic titanium on an industrial scale using such a reaction. If an attempt is made to perform this reaction merely by heating [the reactants] in a high-temperature furnace, it is almost impossible to obtain a sufficient reaction, so that it is naturally not easy to obtain metallic titanium on an industrial scale. In the method of the present invention, on the other hand, titanium tetrachloride and hydrogen are caused to jet from a nozzle into a hydrogen gas [atmosphere], and a current is passed through this [reaction mixture] so that one pole is formed. Another receiving pole is disposed in the bottom portion of the furnace relative to this [first pole], and an electrical discharge is caused to take place between the two poles so that the mixed gas is heated to a temperature of 2000°C or higher; as a result, the titanium tetrachloride and hydrogen readily react. The metallic titanium [that is produced by this reaction] is separated out; meanwhile, the hydrochloric acid and unreacted titanium tetrachloride are conducted elsewhere and appropriately treated. In this way, metallic titanium can be produced continuously by means of a relatively simple apparatus and method.

Initially, when hydrogen alone is caused to jet into the hydrogen gas [atmosphere] from a jet hole, and a discharge is caused to take place between [this pole] and the receiving pole, a dark purple discharge is generated. However, when hydrogen gas mixed with titanium tetrachloride gas is passed through the system, the discharge abruptly changes to a discharge with a white color, thus clearly indicating that a reaction is taking place. If the conditions of the discharge current and discharge distance are [appropriately] satisfied, the heating temperature of the gas can be sufficiently elevated so that this temperature reaches 2000°C, thus making it possible to manufacture the desired metallic titanium.

The [attached] figure shows one example of the working of the method of the present invention. Here, a nozzle 1 is provided which has a jet tube 3 that jets a mixed gas consisting of titanium tetrachloride gas and hydrogen gas into a furnace 4. A hydrogen gas feeding tube 2 is disposed outside the jet tube 3 inside the nozzle 1, and the receiving pole 5 is set at the pole that receives the gas that is caused to jet from the nozzle 1. With the jet nozzle 1 used as one pole and the receiving pole 5 used as the other pole, these poles are connected to a power supply, and when an appropriate voltage is applied, an arc discharge is generated through the mixed gas, so that the mixed gas is heated, and metallic titanium is produced. This system has a structure in which the hydrogen chloride gas produced by the reaction and the excess hydrogen are conducted elsewhere by a discharge pipe 6 and treated.

Thus, it is certain that the present invention provides a method which can be worked by means of a relatively simple apparatus, and which has an industrial value not seen in conventional methods.

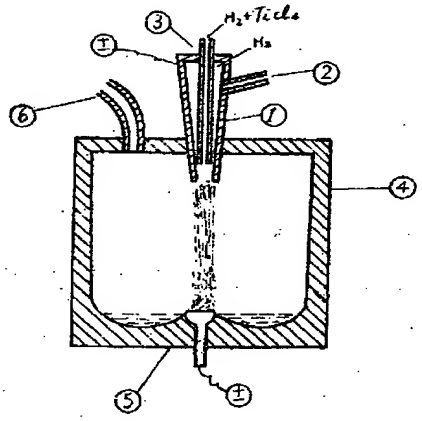
In order to show one example of the working of the method of the present invention, $TiCl_4 + H_2$ was blown into [a reaction vessel] from a tungsten pole 1 with an internal diameter of 5 mm, and a discharge was caused to take place between this pole and a counter-pole with the total amount of H_2 set at approximately 50% in excess of the stoichiometric amount. The various conditions in this case were as follows:

Distance between electrodes	15 mm
Transformer connection	Series
Secondary voltage	3720 V
Secondary current	533 mA
Amount of $TiCl_4 + H_2$	approximately 4 L/min
Gas pressure	320 mm H_2O

A discharged was continuously performed under these conditions. As a result, a branch-form powdered crystal accumulated in the vicinity of the lower electrode. When this product was analyzed, it was found that the product consisting of 89% metallic titanium, with the remainder consisting of lower chlorides. When this product was heated in a vacuum, metallic titanium with [a purity of] 99.4% was obtained.

Claims

A method for manufacturing metallic titanium from titanium tetrachloride by means of an electric discharge, in which a mixed gas consisting of titanium tetrachloride gas and hydrogen is caused to jet [into a reaction vessel] from one pole, and an electrical discharge is caused to take place between this pole and a receiving pole for this [pole], so that metallic titanium is formed, as is described in detail in the main text in accordance with the object described in the main text.



特　許　庁　　特許出願公告

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電気放電による金属チタンの製造法

図面の略解

図面は本発明方法実施の一例を示すものにして其の縦断正面図を示す。

発明の詳細なる説明

本発明は四塩化チタンと水素とを一極より噴出せしめ他極との間に放電を起さしめて四塩化チタンを分解し金属チタンを得ることを特徴とする電気放電により四塩化チタンより金属チタンを得る方法に係り其の目的とする所は従来の例えば金属マグネシウムによる置換法等に比し操作容易に経済的に金属チタンを得る方法を得んとするに在り。

本発明は四塩化チタンを電気放電により金属チタンを得る方法にして四塩化チタンと水素とを一極より噴出せしめ之に対する他極との間に電気放電を起さしめ四塩化チタンを分解し金属チタンを得るものである。

従来金紅石、チタン鉄鉱、二酸化チタン等を塩素瓦斯により処理し四塩化チタンを製造する事は工業化せられ広く採用せられている所である。而して斯くして得たる四塩化チタンより金属チタンを得る方法に関しては近時特に研究せられ種々の方法が提案せられつゝあるも目下工業的に実施せられているものは四塩化チタンと金属マグネシウムとを高温不活性気中にて反応せしめ次式により金属チタンを得る方法である。

$$TiCl_4+2Mg=Ti+2MgCl_2$$

然るに斯る方法に於て高純度の金属マグネシウムを金属チタンに対し1.1乃至1.5倍を必要とするの外、装置複雑にして且つ通常断続的操業を行はざるを得ざる憾あり、之が解決に関し種々研究されつゝある所であるが本発明方法はかゝる従来法の欠点を除き金属チタン製造に極めて斬新的なる方途を寄与せるものである。

即ち四塩化チタンは摂氏2000度以上の高温に於て水素と反応し次式により金属チタンを生ずるものなることに着目し種々研究せる結果本発明を完成せるものである。

$$TiCl_4+2H_2=Ti+4HCl$$

然るにかゝる反応を利用し工業的に金属チタンを得ることは極めて困難なるものにして単にこれを高温炉中にて行はんとするもこの反応を充分行ましむること殆んど不可能にして勿論工業的に金属チタンを得ること容易ならず、然るに本発明方法により水素気中にて四塩化チタンと水素とをノヅルより噴出せしめ之に通電して一極となし之に対し炉の底部に他の受極を設け両者間に電気放電を行はしめ混合瓦斯を2000℃以上に加熱し、四塩化チタンと水素とは容易に反応し金属チタンを分離し、一方塩酸及び未反応四塩化チタンは他に導き之を適宜処置することにより連続的に而かも比較的簡易なる装置並びに方法により金属チタンを得る事可能なるものである。

最初水素気体中に於て水素のみを噴出孔より噴出せしめ受極との間に放電せしむる時は暗紫色の放電を為すも四塩化チタン瓦斯の混合水素瓦斯を通ずる時は明白色を帯びたる放電に急変し明らかに反応を為すことを示す。放電電流と放電距離との条件を満足する時は十分瓦斯の加熱温度を高め2000℃に達せしめ得べく金属チタニウムを製造することが出来る。

図面は本方法実施の一例にして炉4に四塩化チタン瓦斯と水素瓦斯との混合瓦斯を噴出する噴出管3を具備するノヅヅル1を設けノヅヅル1内の噴出管3の外側に水素送入管2を設く、受極5はノヅヅル1より噴出せる瓦斯を受ける極にして、噴出ノヅヅル1を一極とし受極5を他極とし電源に接続し適当なる電圧を加うる時は混合瓦斯を通じアーク放電を生じ混合瓦斯を加熱し金属チタニウムを生成する。反応に依り生じたる塩化水素瓦斯及び余剰水素は排出管6より他に導き処理する構造とする。

本発明は斯くの如く比較的簡易なる装置にて実施し得る方法にして従来其の例を見ざる工業的価値ある方法なりと確信するものである。

本発明方法の実施の一例を示すに、内径5m/mのタングステン極1から$TiCl_4+H_2$を吹き込みこの際全体のH_2の量が化学当量の約50%増になる様にして対極との間に放電を行はせた。この際の諸条件は次の如くである。

電弧間隙	15m m
トランス接続	直　列
2次電圧	3720V
2次電流	533m A.
$TiCl_4+H_2$量	約4 L./分
瓦斯圧	320m m水柱

この条件の下で放電を継続した所が下の電極附近に樹枝状の粉状の結晶が堆積したこれを分析せるに、金属チタニウム89%で他は低級塩化物であつたのでこれを真空加熱せるに、99.4%の金属チタニウムが得られた。

特許請求の範囲

本文所載の目的に於て本文に詳記せる如く四塩化チタン瓦斯と水素との混合瓦斯を一極より噴出し之に対する受極との間に電気放電を起さしめて金属チタンとなす四塩化チタンより電気放電により金属チタンを製造する方法。